NACIONALNI FORENZIČNI LABORATORIJ
NATIONAL FORENSIC LABORATORY

Vodovodna 95
1000 Ljubljana
SLOVENIJA

T: +386 (0)1 428 44 93
E: nfl@policija.si
www.policija.si

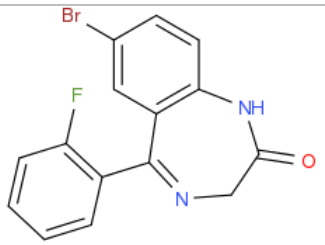
ANALYTICAL REPORT

Flubromazepam (C15H10BrFN2O)

7-bromo-5-(2-fluorophenyl)-1,3-dihydro-2H-1,4-benzodiazepin-2-one

Remark – other NPS detected: none

Sample ID:	1192-15
Sample description:	powder - white
Sample type:	test purchase (RESPONSE -purchasing)
Comments[1]:	
Date of entry:	10/5/2015

Substance identified-structure[2] (base form)	
Systematic name	7-bromo-5-(2-fluorophenyl)-1,3-dihydro-2H-1,4-benzodiazepin-2-one
Other names	
Formula (per base form)	C15H10BrFN2O
M_w (g/mol)	333,15
Salt form	base
StdInChIKey	ZRKDDZBVSZLOFS-UHFFFAOYSA-N
Compound Class	Benzodiazepines
Other NPS detected	none
Add.info (purity..)	

[1] This report has been produced with the financial support of the Prevention of and fight against crime Programme of the European Union (grant agreement number JUST/2013/ISEC/DRUGS/AG/6413). The contents of this report are the sole responsibility of the National Forensic Laboratory and can in no way be taken to reflect the views of the European Commission.

[2] Created by OPSIN free tool: http://opsin.ch.cam.ac.uk/ **DOI:** 10.1021/ci100384d

Report updates

date	comments (explanation)

Instrumental methods (if applied) in NFL

1. GC-MS (Agilent): GC-method is RT locked to tetracosane (RT=9.53 min). Injection volume 1 ml and split mode (1:50) . Injector temperature: 280 ^{0}C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickens 0.25 mm. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 ^{0}C for 1 min, followed by heating up to 293 ^{0}C at a rate of 18 ^{0}C/min, hold for 6.1 min, than heating at 50 ^{0}C/min up to 325 ^{0}C and finally 2.8 min isothermal. MSD source EI = 70 eV. GC-MS transfer line T= 235^{0}C, source and quadropole temperatures 280^{0}C and 180^{0}C, respectively. Scan range m/z scan range: from 50 (40) to 550 amu.

2. HPLC-TOF (Agilent): 6230B TOF with Agilent 1260 Infinity HPLC with binary pump, column: Zorbax Eclipse XDB-C18, 50 x 4.6 mm, 1.8 micron. Mobile phases (A) 0.1% formic acid and 1mM ammonium formate in water; (B) 0.1% formic acid in methanol (B). Gradient: starting at 5% B, changing to 40% B over 4 min, then to 70% over 2 min and in 5 min to 100%, hold 1 min and back to 5%, equilibration for 1.7 min. The flow rate: 1.0 ml/min; Injection volume 1 µl. MS parameters: 2GHz, Extended Dynamic range mode to a maximum of 1700 amu, acquisition rate 1.30 spectra/sec. Sample ionisation: by Agilent Jet Stream technology (Dual AJS ESI). Ion source: positive ion scan mode with mass scanning from 82 to 1000 amu. Other TOF parameters: drying gas (N2) and sheath temperature 325 °C; drying gas flow rate 6 l/min; sheath gas flow rate 8 l/min; nebulizer 25 psig; Vcap. 4000 V; nozzle 2000 V; skimmer 65 V; fragmentor 175 V and Octopole RF 750 V. 3. FTIR-ATR (Perkin Elmer): scan range 4000-400 cm^{-1}; resolution 4 cm^{-1}

3.FTIR-ATR (Perkin Elmer): scan range 4000-400 cm-1; resolution 4cm-1

4. GC- (MS)-IR condensed phase (GC-MS (Agilent) & IR (Spectra analyses-Danny)
GC-method: Injection volume 1 ml and split mode (1:5). Injector temperature 280 ^{0}C. Chromatographic separation as above **(1).** Split MS : IR = 1:9.
MSD source EI = 70 eV. GC-MS transfer line T= 235^{0}C, source and quadropole temperatures 280^{0}C and 180^{0}C, respectively. Scan range m/z scan range: from 50 (40) to 550 amu.
IR (condesed phase): IR scan range 4000 to 650, resolution 4 cm^{-1} .

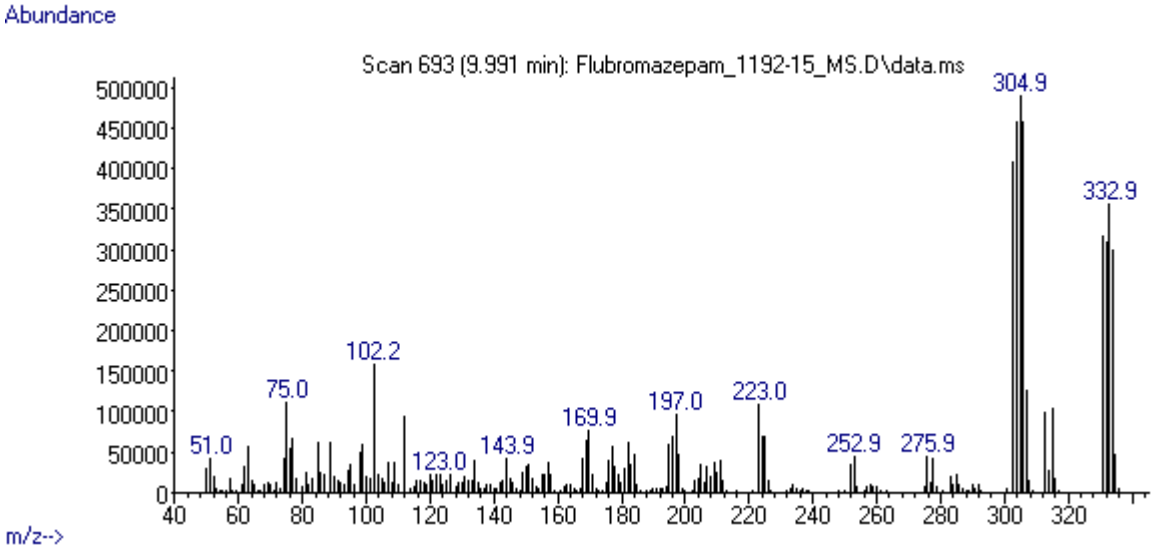
Supporting information

Solubility in	result/remark
CH_2Cl_2	soluble
MeOH	soluble
other	not tested

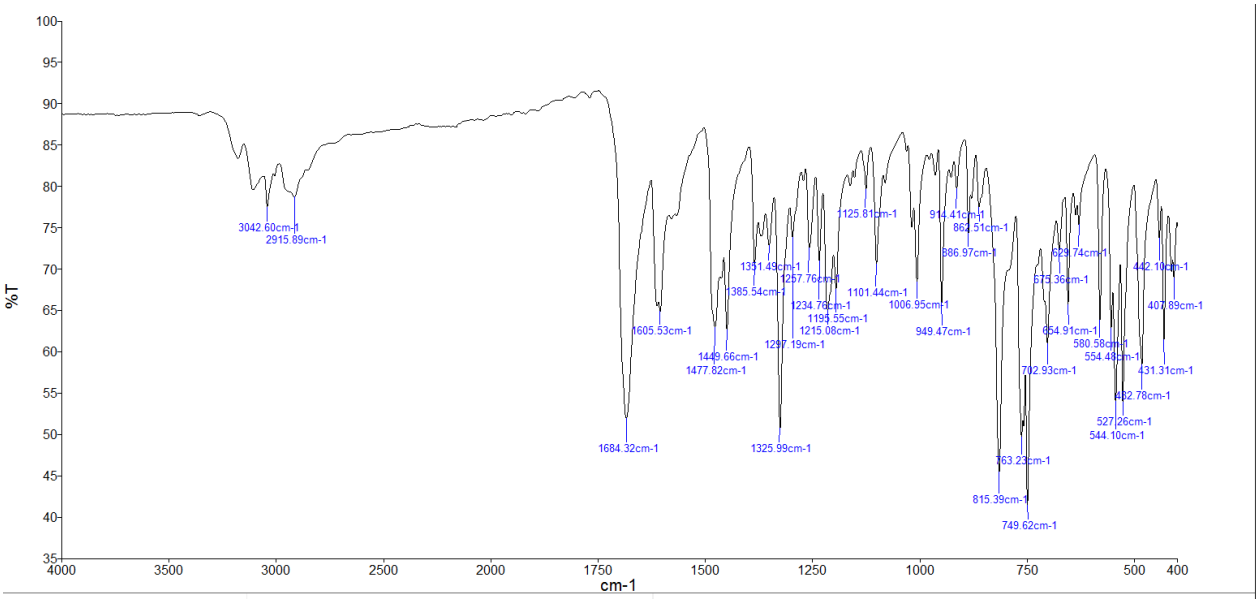
Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 9,99 BP(1): 304; BP(2): 303,BP(3) :305, extr. CH2Cl2
HPLC-TOF	+	Exact mass (theoretical): 331,9961; measured value Δppm:-0,03; formula:C15H10BrFN2O
FTIR-ATR	+	direct measurement
FTIR (condensed phase) always as base form	+	ex. CH2Cl2
HPLC-TOF	+	Exact mass (theoretical): 331,9961; measured value Δppm:-0,03; formula:C15H10BrFN2O
NMR	+	
validation		
other		pure by GG, HPLC, NMR

ANALYTICAL RESULTS

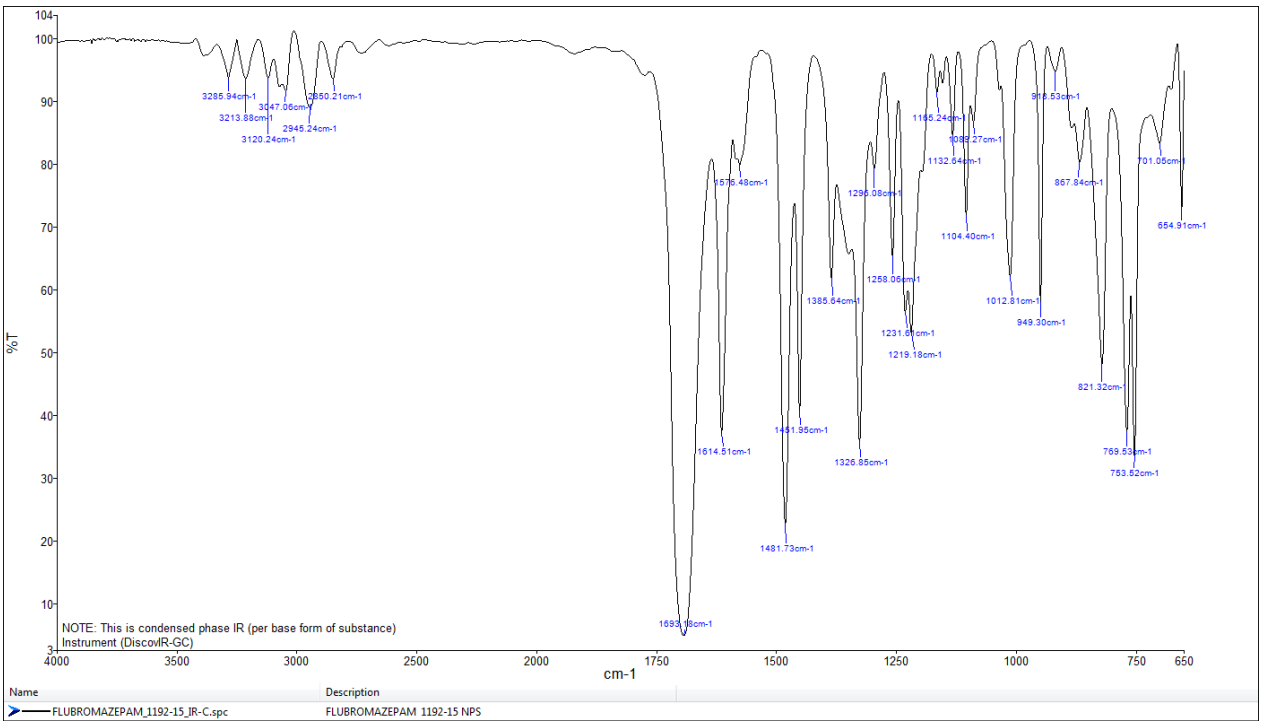
MS (EI)



FTIR-ATR



IR (condensed phase)



Target Compound Screening Report

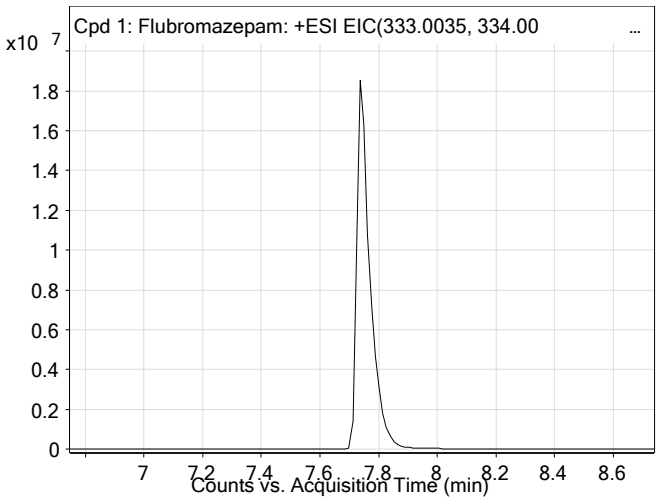
Data File	Flubromazepam_1192-15_TOF.d	**Sample Name**	Flubromazepam
Sample Type	Sample	**Position**	P1-D2
Instrument Name	6230B TOF LC-MS	**User Name**	
Acq Method	droge general-13-5-2015-XDB-C18-ESI-poz.m	**Acquired Time**	7/27/2015 10:38:51 AM
IRM Calibration Status	Success	**DA Method**	Droge_Default.m
Comment	extract in MeOH		

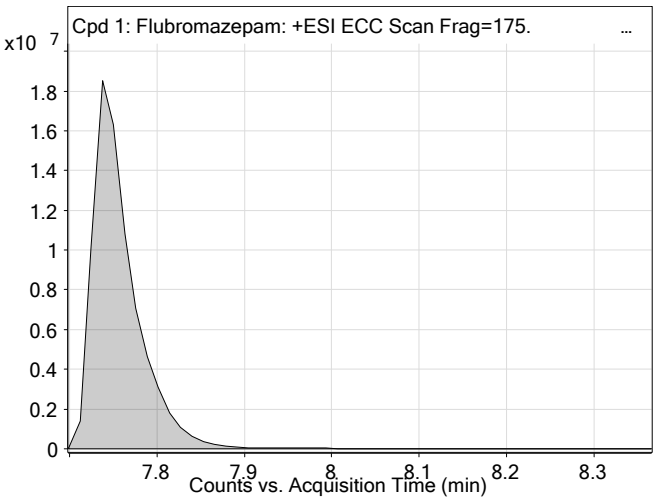
Compound Table

Label	Tgt Name	MFG Formula	Obs. RT	Obs. Mass
Cpd 1: Flubromazepam	Flubromazepam	C15 H10 Br F N2 O	7.744	331.9961

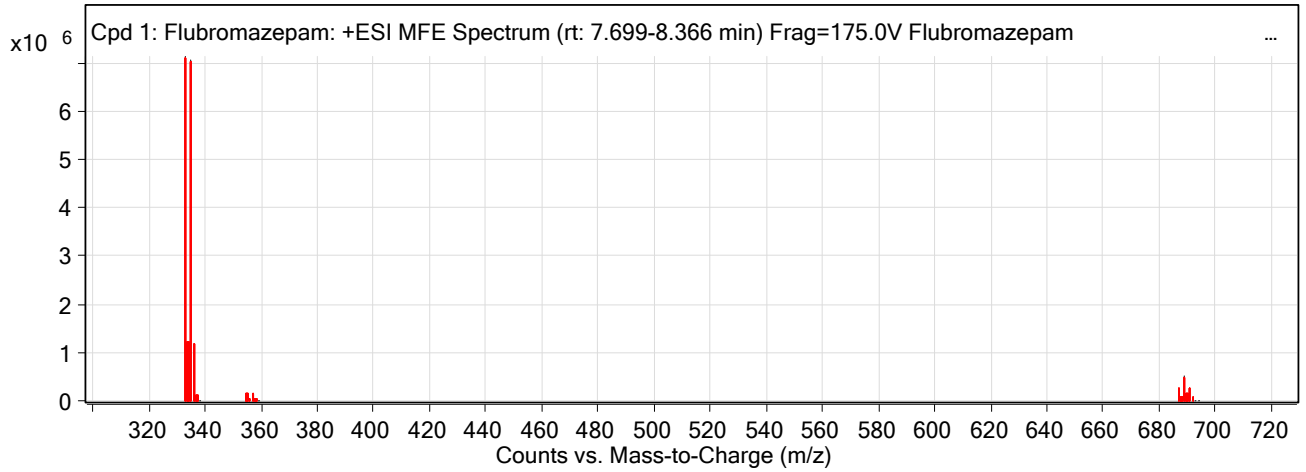
Name	Obs. *m/z*	Obs. RT	Obs. Mass	DB RT	DB Formula	DB Mass	DB Mass Error (ppm)	Find Cpds Algorithm
Flubromazepam	333.0033	7.744	331.9961	7.74	C15 H10 Br F N2 O	331.9961	-0.03	Find by Molecular Feature

Compound Chromatograms





MFE MS Zoomed Spectrum

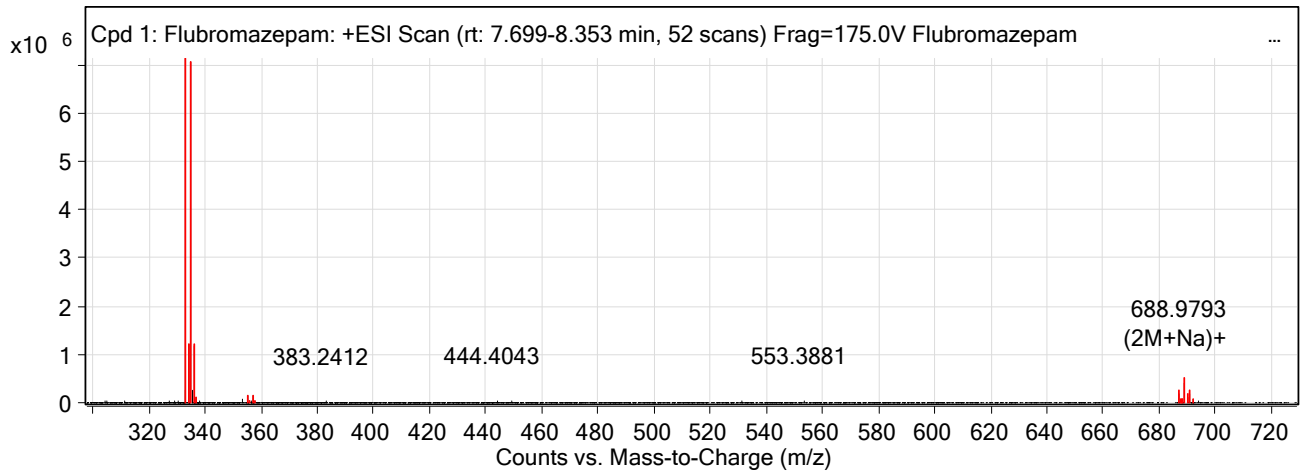


MS Spectrum Peak List

Obs. *m/z*	Charge	Abund	Formula	Ion/Isotope

333.0033	1	7135505.5	C15 H10 Br F N2 O	(M+H)+
334.0069	1	1185309.57	C15 H10 Br F N2 O	(M+H)+
335.0013	1	7059902.39	C15 H10 Br F N2 O	(M+H)+
336.0048	1	1139577.94	C15 H10 Br F N2 O	(M+H)+
354.9852	1	163052.95	C15 H10 Br F N2 O	(M+Na)+
356.9835	1	162900.45	C15 H10 Br F N2 O	(M+Na)+
686.981	1	239342.13	C15 H10 Br F N2 O	(2M+Na)+
688.9796	1	507278.22	C15 H10 Br F N2 O	(2M+Na)+
689.9821	1	158608.16	C15 H10 Br F N2 O	(2M+Na)+
690.978	1	256964.89	C15 H10 Br F N2 O	(2M+Na)+

MS Zoomed Spectrum



--- End Of Report ---

University
of Ljubljana

Faculty *of Chemistry and Chemical Technology*

Večna pot 113
P. O. Box 537
SI-1001 Ljubljana
Slovenia
Phone: +386 1 479 8558
janez.kosmrlj@fkkt.uni-lj.si

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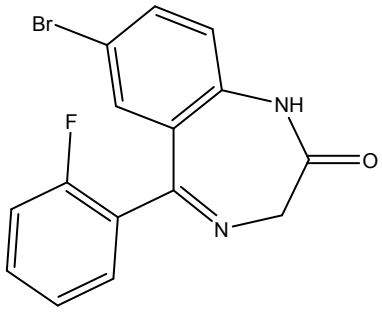
REPORT

Sample ID:	**1192-15**
Our notebook code:	P-1192-15
NMR sample preparation:	15 mg dissolved in 0.7 mL $CDCl_3$
NMR experiments:	^1H, ^{13}C, ^1H–^1H *gs*-COSY, ^1H–^{13}C *gs*-HSQC, ^1H–^{13}C *gs*-HMBC, ^1H–^{15}N *gs*-HMBC.
Proposed structure with chemical name:	 7-bromo-5-(2-fluorophenyl)-1,3-dihydro-2*H*-benzo[e][1,4]diazepin-2-one
Comments:	- Structure elucidation based on 1D and 2D NMR spectra - Compound is pure by NMR
Supporting information:	Copies of ^1H and ^{13}C NMR spectra
Author:	Prof. Dr. Janez Košmrlj, Doc. Dr. Krištof Kranjc
Date of report:	September 30, 2015

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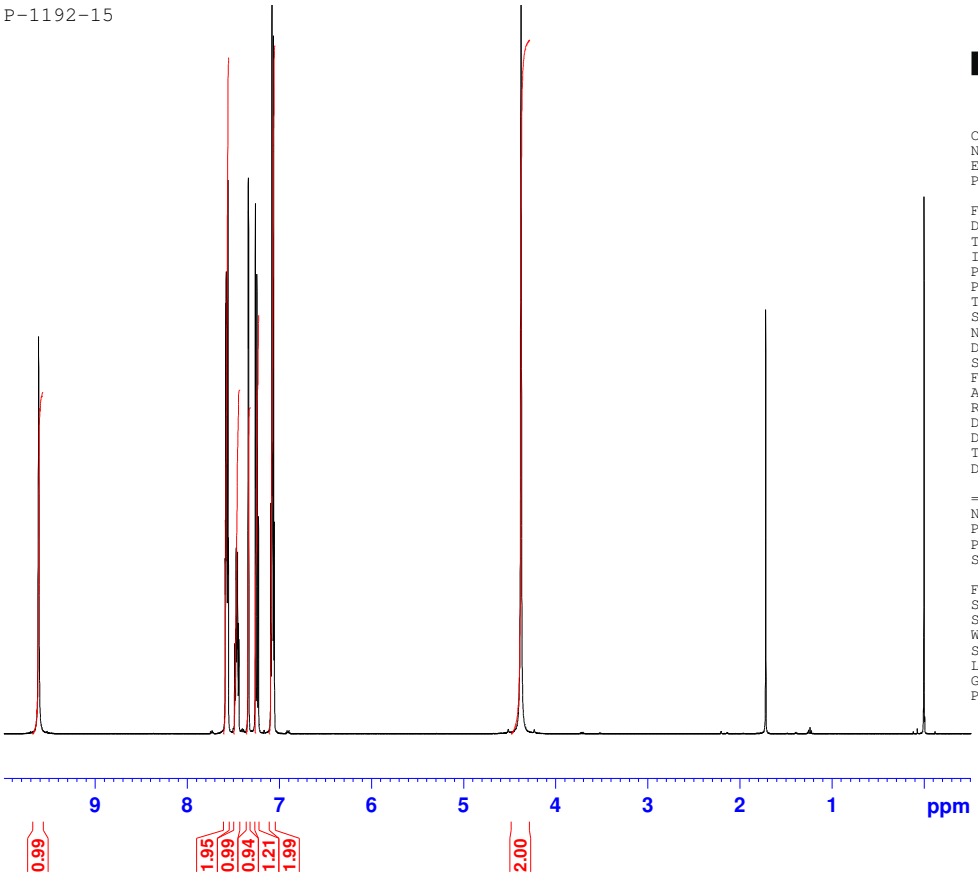
P-1192-15



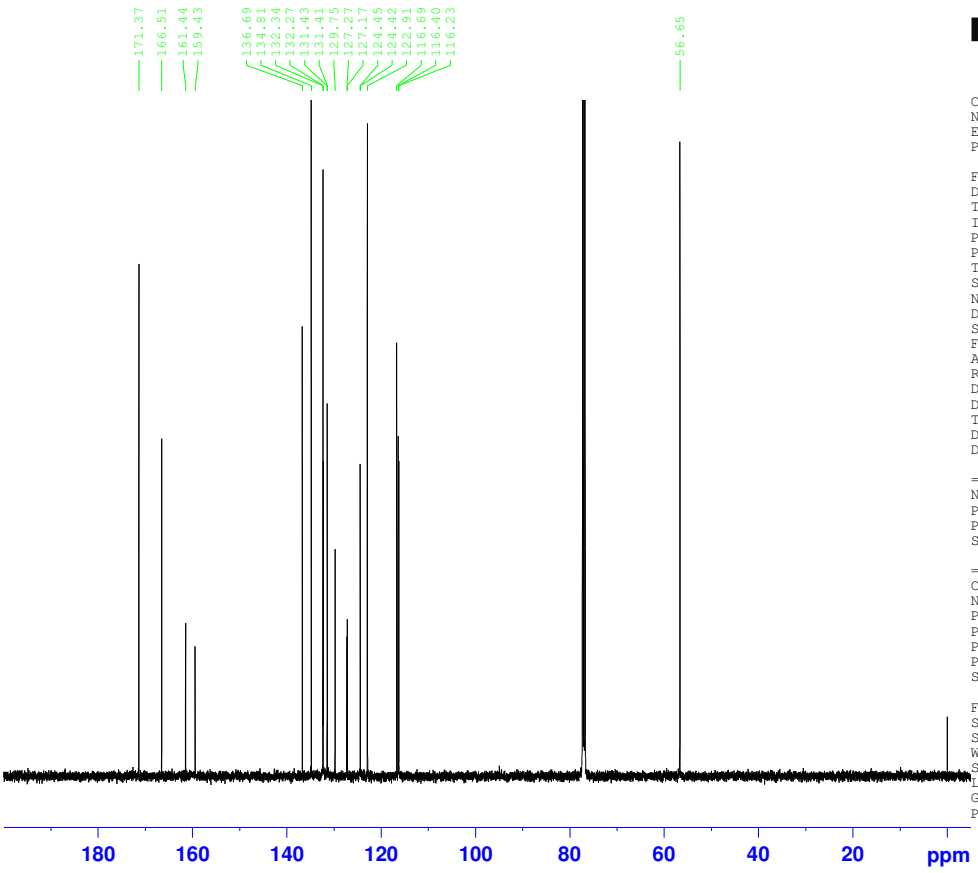

```
Current Data Parameters
NAME          P-1192-15
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20150815
Time               4.39
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDC13
NS                   16
DS                    2
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ            3.1719923 sec
RG                  128
DW               48.400 usec
DE                6.50 usec
TE                298.1 K
D1           1.00000000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                8.90 usec
PLW1        26.00000000 W
SFO1        500.1330885 MHz

F2 - Processing parameters
SI                65536
SF          500.1300117 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

P-1192-15




```
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EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
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Time               6.42
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDC13
NS                 3072
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ            1.1010548 sec
RG                 2050
DW               16.800 usec
DE                6.50 usec
TE                299.0 K
D1           1.00000000 sec
D11          0.03000000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                9.00 usec
PLW1       122.00000000 W
SFO1        125.7703637 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2             80.00 usec
PLW2        26.00000000 W
PLW12        0.32179001 W
PLW13        0.20595001 W
SFO2        500.1320005 MHz

F2 - Processing parameters
SI                32768
SF          125.7577899 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```